Functional Materials

LBP.MS.P16
Investigating dislocation activity in Nb_2AlC layered carbide using In situ TEM nanomechanical testing

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Keywords: Nb_2AlC, Dislocation, In situ TEM

MAX phase layered crystals have attracted much interest in scientific community because of their damage tolerance (up to ~25% dissipation energy) and high oxidation resistance (up to 1300°C), making them a promising material system for high temperature applications.

In these layered materials - unlike many crystals - dislocation activity is believed to be partially reversible and is only limited to easy slip systems along basal planes. In the past, fully reversible, rate independent and closed hysteresis loops are observed in cyclic stress-strain curves which are attributed to formation and annihilation of incipient kink bands (IKBs) and dislocation walls [1].

To date, the precise mechanism of dislocation wall and IKBs nucleation is not known and no decent investigation has been carried out to understand their dynamics using In situ TEM. It is believed that by controlling the parameters which influence these dynamics, it is possible to control the mechanical properties dramatically.

In this study, a wedge shaped thin foil of polycrystalline Nb_2AlC was prepared by special mechanical grinding and ion milling procedure. The edge of the sample was cut using Focused Ion Beam (FIB) to reduce bending of thin parts during In situ nanoindentation. Convergent beam electron diffraction (CBED) technique was used to find the suitable crystal orientation and nanomechanical testing was performed using the Hysitron® Picoindenter set up with a cube-corner indenter inside a Phillips CM30 TEM. Indentations were carried out using displacement-time mode and the corresponding force-displacement curve was collected.

Figure 1(a-e) depicts some typical dislocations inside the undeformed Nb_2AlC specimen. Tilting into two beam conditions around <0001> zone axis together with contrast analysis revealed that the dislocations lie in basal plane with 1/3<11-20> type burgers vectors. This is in agreement with previous results published by Farber et al. for another family of MAX phases (Ti_3SiC_2) [2].

Figure 2 shows one in situ TEM nanoindentation experiment for grain oriented near to <0001> direction relative to the electron beam. The blue curve in Figure 2(a) is the displacement-time and the red curve is the corresponding load-displacement function. Before the indentation, the area was pre-characterized to prove that it is dislocation free (Figure 2(b)). It can be clearly seen that basal plane dislocations nucleate and move during the loading in a same slip system without cross-slip or entanglement, confirming that these dislocations are mobile at room temperature as previously proposed by Farber et al. [2]. Lost data points in displacement-time curve during unloading as well as negative values in force-displacement curve are related to adhesion between the tip and the specimen. Discontinuities in force-displacement curve pointed by an arrow in Figure 2(a) are attributed to dislocations formation. Similar behaviour is reported by Molina-Aldareguia et al. [3] for kink band formation during Ex situ indentation on Ti_3SiC_2. Ex situ diffraction contrast analysis was carried out to further characterize the dislocations. As illustrated in Figure 2(d and e), tilting into different two beam conditions revealed that the lines are dislocations with identical burgers vector as invisibility criterion suggests.

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4. The authors would like to acknowledge the research training group 1229 for financial support. Assistances of Christel Dieker and Hana Stara during FIB preparation are also appreciated.

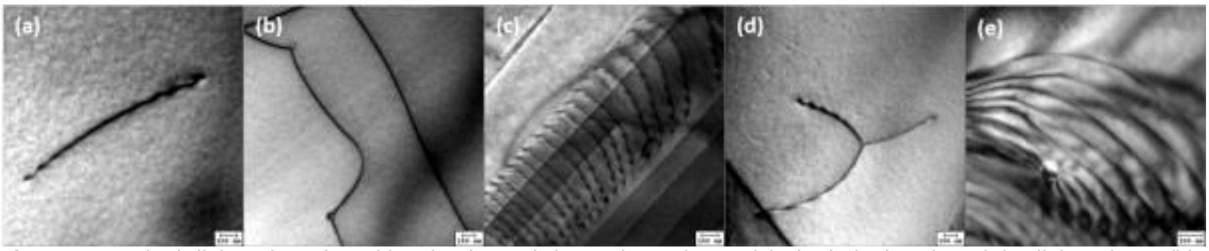

Figure 1. Typical dislocations found in Nb_2AlC undeformed specimen: (a) single isolated straight dislocations, (b) curved dislocations, (c) dislocation walls and arrays, (d) dislocation knots and (e) curved parallel dislocations

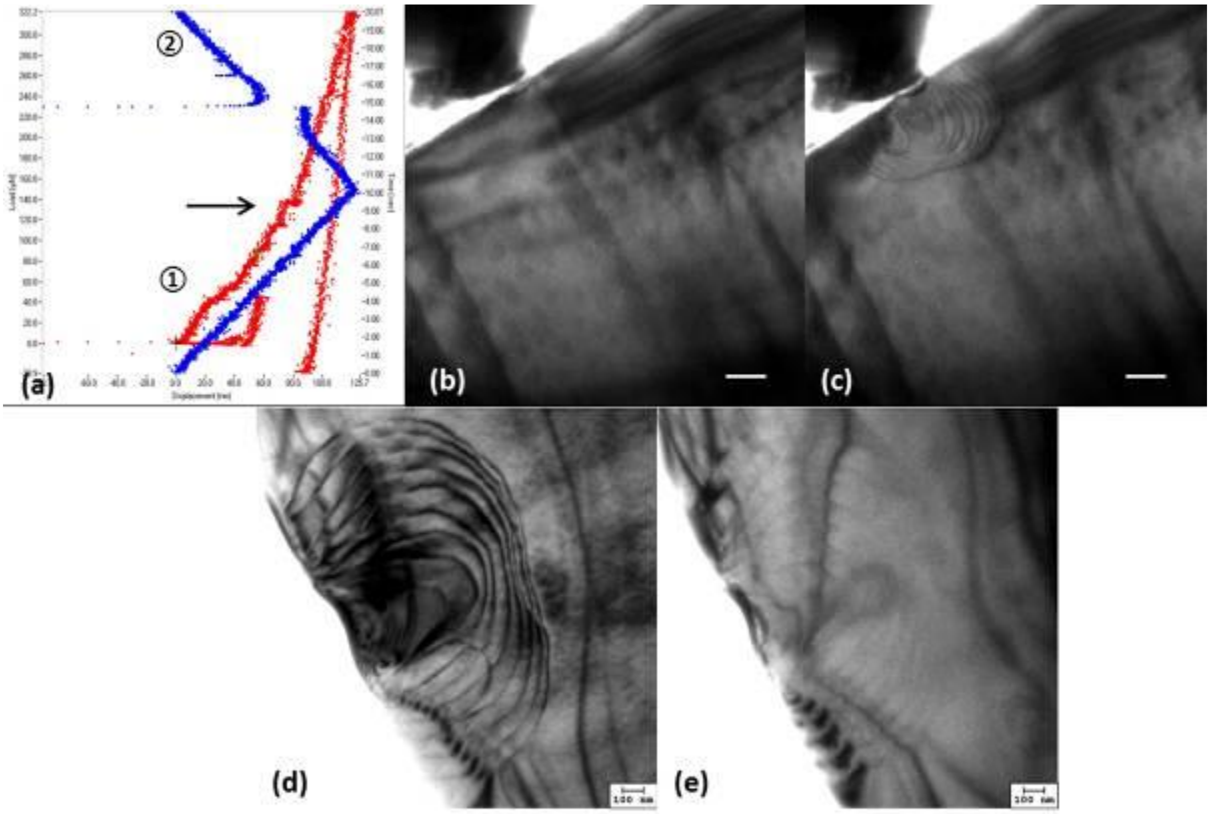

Figure 2. In situ TEM indentation near <0001> direction. (a) load-displacement (red) and displacement-time (blue), (b) start of indentation snapshot corresponding to position indicated with number 1 in (a), (c) end of indentation snapshot corresponding to position indicated with number 2 in (a), (d-e) Ex situ investigation of the same area with higher magnification and imaged with g=[11-20] in (d) and g=[2-1-10] in (e). Dislocations are visible in (d) and invisible in (e), meaning the same type burgers vector. The scale bars in figure (b) and (c) are 500 nm.